EXPLORATORY DEVELOPMENT OF VHF (VERY HIGH FREQUENCY) QUARTZ CRYSTAL RESONATORS(U) PIEZO TECHNOLOGY INC ORLANDO FLA R C SMYTHE ET AL JUL 86 RADC-TR-86-60 F19628-83-C-0061 F/G 9/1 AD-A172 879 1/1 UNCLASSIFIED NL



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RADC-TR-86-60 Final Technical Report July 1986



EXPLORATORY DEVELOPMENT OF VHF QUARTZ CRYSTAL RESONATORS

Piezo Technology, Inc.

Robert C. Smythe and John R. Hunt



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l Introduction

1.1 Goals

The purpose of this program is the exploratory development of VHF and UHF AT-cut quartz crystal resonators utilizing high fundamental frequencies. Specific program objectives are the fabrication of fundamental mode resonators at 70, 100 and 150 MHz and third overtone resonators at 250 and 450 MHz. These objectives have been met. In addition the feasibility of using wet chemical etching to fabricate AT-cut resonators at fundamental frequencies up to 1.6 GHz has been demonstrated.

1.2 Applications and Benefits

The generation of high frequencies is required in a wide range of military (and commercial) VHF, UHF, and microwave systems including communications, navigation, radar, and next-generation high speed logic systems. High frequencies may be generated either directly or by frequency multiplication.

The direct generation of high frequencies offers well known advantages over frequency multiplication. First, direct generation is simpler, resulting in reduced size, weight, cost, and power consumption [1]. Second, since frequency multiplication increases phase noise by 6 dB each time the frequency is doubled, direct generation allows improved phase noise to be obtained [2]. The availability of higher frequency resonators would in some instances allow frequency multiplication to be replaced with direct generation and in other instances would reduce the multiplication required.

Both bulk wave and SAW resonators have uses in high frequency generation. SAW resonator advantages are maximum frequency range, power handling ability, and potentially low cost in high volume applications, due to batch processing.

High frequency bulk wave resonators offer advantages over SAW resonators with regard to aging, temperature stability, vibration sensitivity, and acoustic noise sensitivity. In addition, due to better 0, capacitance ratio (r), and figure of merit (M = Q/r), bulk wave resonators afford circuit simplicity, improved phase noise, and high pullability.

2 Technical Discussion

2.1 Ring-Supported Resonators

A limitation in the fabrication of conventional high frequency bulk wave resonators is that the frequency is inversely related to the wafer thickness. Thus, a 100 MHz AT-cut resonator has a thickness of only 16.6 microns. Such a thin wafer is not only somewhat fragile but also impractical to fabricate by conventional means, which are limited to thicknesses of 30 to 35 microns (approximately 50 MHz for AT-cut resonators) in the best current practice known to us.

To overcome such limitations ring-supported thickness-shear resonators were proposed by Guttwein, Ballato, and Lukaszek [3] and others. Figure 1 shows the essential features of the ring-supported structure. An outer ring provides a strong frame for an integral diaphragm of the required thickness. The mass of the electrodes confines acoustic energy to the central portion of the diaphragm so that the presence of the ring does not affect the resonance. Because the ring and diaphragm are a single homogeneous piece, it is possible to obtain the same frequency-temperature characteristic as for a conventional resonator.

As an alternative to high fundamental frequencies, odd-order overtones of a lower fundamental may be utilized. A limitation is that the resonator impedance level increases as the cube of the overtone; consequently, practical applications are usually restricted to the first few overtones. In the following discussion, while we speak of high fundamental resonators, it will be understood that the overtone modes are also of interest.

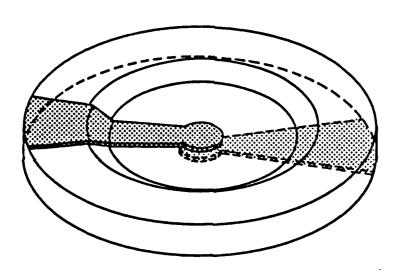


FIG. 1. RING-SUPPORTED RESONATOR

2.2 Fabrication

While the advantages of the monolithic support-ring concept are obvious, fabrication difficulties have limited its exploitation. Methods which may be considered for forming the diaphragm in a suitably lapped and polished wafer include:

- 1) Ion milling (reactive and non-reactive)
- 2) Reactive plasma etching
- 3) Chemical etching.

Argon ion milling for this application has been actively pursued in France for several years. Berte [4], [5] successfully fabricated ring-supported resonators and monolithic filters using non-reactive ion milling to form the diaphragm, and ring-supported resonators, oscillators, and filters are now commercially available in France [6], [7]. AT-cut resonators with fundamental frequencies to 600 MHz have been made [8], and AT-cut fundamentals up to at least 200 MHz are offered commercially. BT- and SC-cuts are also available, as well as X-cut lithium tantalate resonators.

Figure 2 indicates the principle of ion milling. A beam of ions strikes the work with high kinetic energy, removing material from its surface upon impact. By suitably choosing the angle of incidence and at the same time rotating the work, surface irregularities tend to be reduced rather than accentuated. The milling rate is slow - a few microns per hour - and consequently expensive. A 10 cm. ion beam typically requires 8 hoursto mill a load of about 50 resonators.

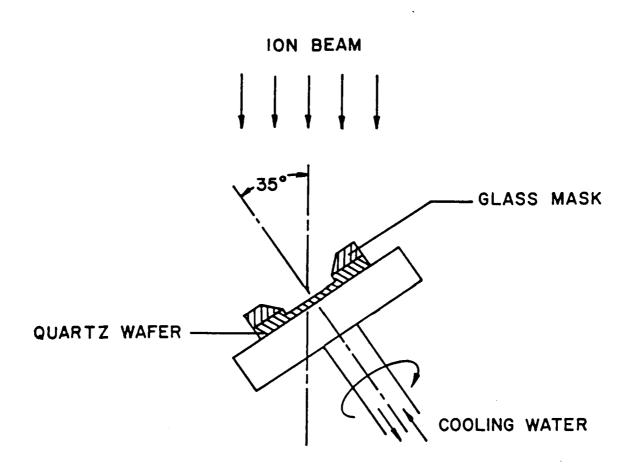


FIG. 2. FIXTURE ARRANGEMENT FOR ION MILLING OF RING-SUPPORTED RESONATOR (AFTER BERTE)

Work has been done at TRW [9] using an ion beam composed of a mixture of non-reactive argon gas and reactive perfluoroethane gas. This gas mixture is said to eliminate undesirable trenching and redeposition observed along the edges of the wells formed with argon ion milling alone. Utilizing this technique, etch rates as high as 30 microns per hour have been reported and devices fabricated with fundamental frequencies of 200 MHz.

Reactive plasma etching is widely used in semiconductor processing. Limited work at PTI prior to this program indicates that moderately high stock removal rates can be obtained; however, there was considerable evidence of surface damage. Figure 3 shows the planar etching system used. Because the etch rate is fairly high and a large number of wafers can be etched simultaneously, plasma etching is potentially a low-cost process, but requires further development.

Chemical etching has been used for many years in the crystal industry to remove wafer damage caused by mechanical lapping. New insight into etching has been provided by the work of Vig and co-workers [10], who introduced the depletion-layer concept to explain improvements in surface roughness which they observed. They speculated that chemical etching might be used to fabricate the ring-supported resonator wafer.

The feasibility of using chemical milling to form the ring-supported structure was demonstrated at PTI prior to the present program. Nevertheless, it was not certain whether all program objectives could be met using the process. Therefore, it was initially planned to investigate not only chemical milling but also plasma etching and ion milling. The favorable results obtained with chemical milling, however, and the relative simplicity of the technique, made it advisable to concentrate on it to the exclusion of the other processes.

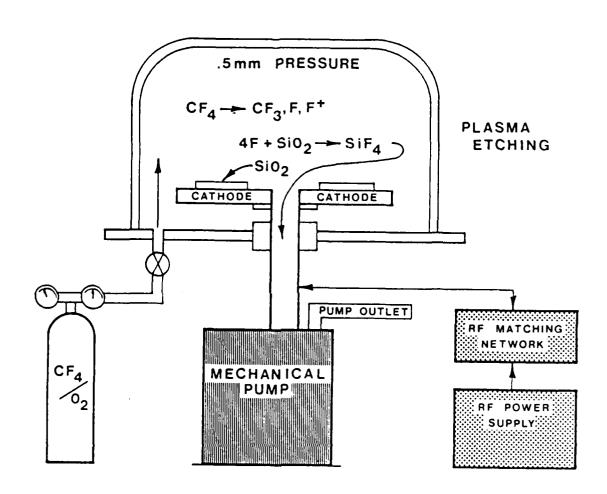
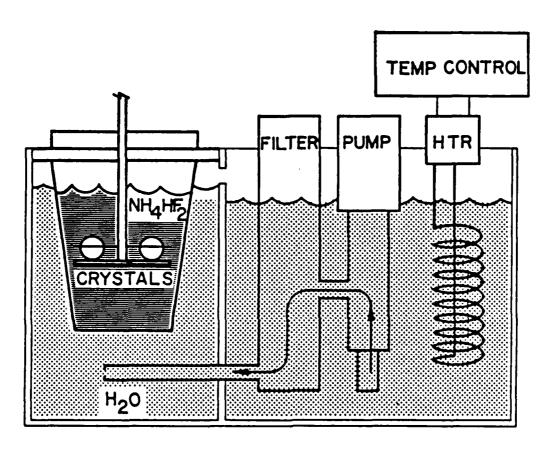


FIGURE 3. PLANAR REACTIVE PLASMA ETCHING SYSTEM

Figure 4 shows an ammonium bifluoride etch system. Experimentally determined etch rates are shown in Figure 5 for saturated ammonium bifluoride and AT-cut quartz.

The complete resonator process sequence, indicated in simplified form in Figure 6, involves a number of additional steps. Our investigations have, of necessity, concentrated on the development of methods for forming the ring-supported structure (diaphragm formation). Key to this are the wafer lapping and polishing operations. Plasma etching, chemical etching, and ion milling all require high quality wafers as starting material. In particular,

- 1) The parallelism of the wafers must be excellent, since it will not be improved by the etching or milling processes. Departures from parallelism affect motional parameters and unwanted mode performance and may degrade Q.
- 2) The surfaces should have low damage. Certain lapping/polishing sequences can yield surfaces which, while apparently highly polished, will, upon etching, reveal large amounts of damage, with consequent Q degradation.
- 3) The starting material natural or cultured quartz must be of adequate quality. The requirements imposed by the ion milling and plasma etching processes are not known, but for chemical etching a limitation is "etch channels" [10] capillary passages in the wafer induced by the etching process. Present-day electronic grade and premium grade cultured quartz is inadequate for chemical etching because of high channel density. Improvements may be obtained by sweeping and by improved growth methods. Much natural quartz possesses adequately low etch channel density. Cultured quartz swept by PTI also showed very low etch channel densities.



AMMONIUM BIFLUORIDE ETCH

$$SiO_2 + 2 H_2O \implies Si(OH)_4$$

 $Si(OH)_4 + 6 HF \implies 2 H^+ + SiF_6^- + 4 H_2O$

FIG. 4. AMMONIUM BIFLUORIDE ETCH SYSTEM,

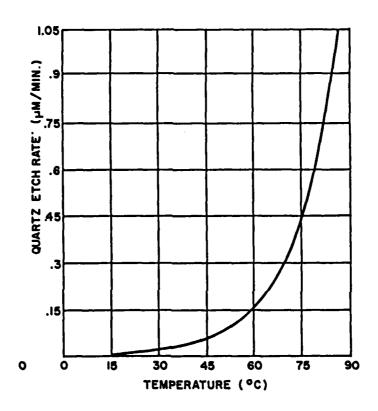


FIG. 5. ETCH RATE VS. TEMPERATURE SATURATED NH4HF2

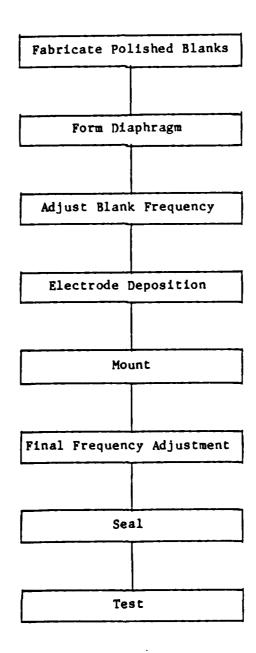


Figure 6. Simplified Process Sequence

In the chemical etching process the use of unpolished blanks may be considered, as etching removes material damaged by lapping and at the same time reduces surface roughness. There are two limitations:

- 1) Surface finish may not be adequate to obtain desired Q.
- 2) Parallelism obtained in lapping is generally inferior to that obtained by polishing, with the consequences mentioned earlier. The parallelism of unpolished blanks cannot be accurately measured by standard techniques. On the other hand, the parallelism of polished blanks can easily be measured by observing self-interference fringes under monochromatic light.

Despite these restrictions, the use of carefully prepared, unpolished blanks cannot be ruled out in all instances.

2.3 Experimental Results

Using chemical milling with ammonium bifluoride, resonators were fabricated at frequencies from 70 MHz to 1.6 GHz. The work began with 70 MHz units and proceeded, for the most part, in order of increasing fundamental frequency.

Resonator parameter measurements were made using an automatic measurement system utilizing the H-P 4191A Impedance Analyzer [11]. Mode plots employed either an H-P 140 series spectrum analyzer/tracking generator, an H-P 3577A network analyzer, or a Polarad ZPV vector analyzer in conjunction with an H-P 8662A synthesized signal generator.

2.3.1 70 MHz Fundamental Resonators

The first 70 MHz units had a blank diameter of 0.25 inch and aluminum electrodes 40 mils in diameter, were mounted in PTI type "F" enclosures (similar to HC-18), and sealed in dry nitrogen atmosphere. Figure 7 is a mode plot for one of these units, while Table 1 presents the equivalent circuit parameter values for the fundamental mode and two unwanted responses.

The electrode diameter of 40 mils was used initially in order to make use of an available mask. Subsequently, a 20 mil mask was fabricated, and additional units were made having 20 mil electrodes, also sealed in a dry nitrogen atmosphere. Table 2 contains measured parameter data. Figure 8 is a mode plot for one of these units and is fairly typical, although there is considerable unit-to-unit variation. Decreasing the electrode diameter increased the spacing between the fundamental and anharmonic modes, as

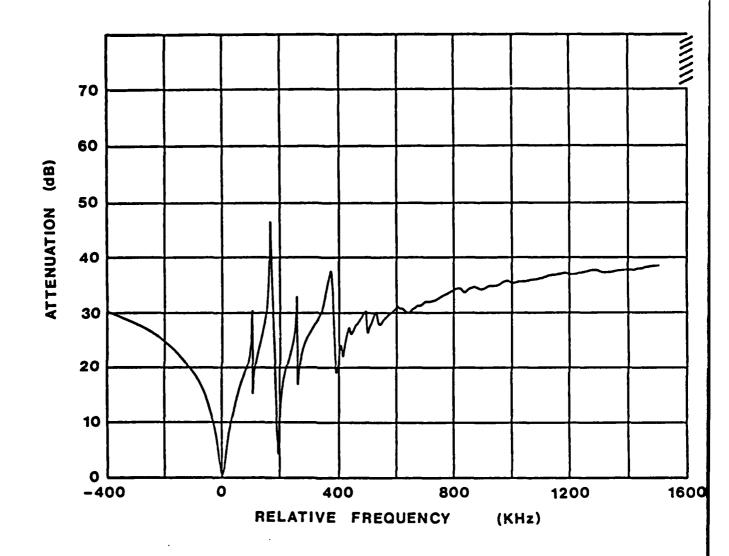


FIGURE 7. MODE PLOT, UNIT NO. 20, 68.2528 MHz

TABLE 1
EQUIVALENT CIRCUIT PARAMETERS
UNIT NO. 20

MODE	f _s (kHz)	R ₁ (Ohms)	C ₁ (fF)	C _O (pF)	(x 10 ³)
FUND.	68252.81	20.8	6.85	2.08	16
SPUR 1	68363.38	476	0.15		34
SPUR 2	68455.27	74.7	1.03		30

TABLE 2
RESONATOR PARAMETER DATA

Unit No.	Fs (kHz)	R1 (Ohms)	I (mA.)	L1/C1 (mH/fF)	CO/r (pF/-)	kQ/m
71	71873.862	40.68	0.496	2.46 1.9905	1.01 507	
72	71315.053	30.79	0.557	2.13 2.3400		
73	71212.093	26.57	0.588	1.99 2.5053		
75	67778.291	30.24	0.561	2.32 2.3750		33 79
77	71209.215	33.15	0.541		1.01 406	
78	71108.334	30.83	0.557	2.04 2.4539		
79	72957.629	40.45	0.498		1.02 523	
80	69303.029	49.81	0.451	2.95 1.7855	0.99 55 7	26 46

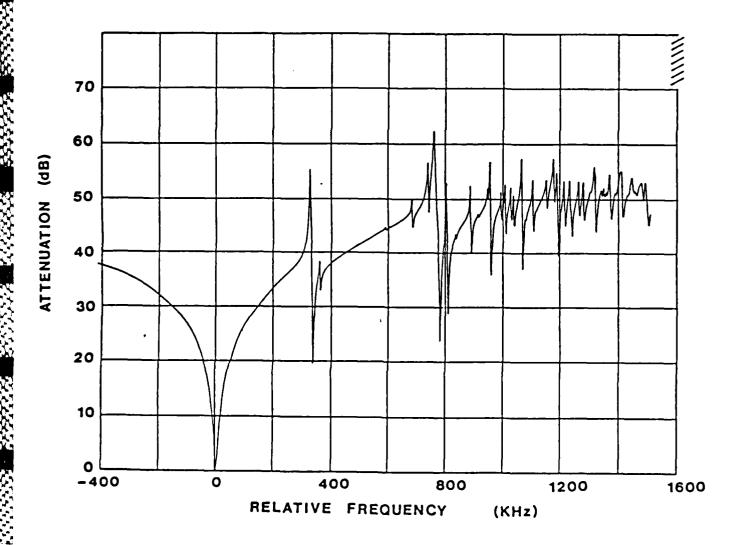


FIGURE 8. MODE PLOT, UNIT NO. 78, 72.9576 MHz

predicted by trapping theory. The first unwanted mode was typically more than 300 kHz above the fundamental mode. This is an antisymmetric mode, but is weakly excited due to slight unavoidable asymmetry of the resonator structure. There was also an increase in Q from an average of 15 thousand to 30 thousand, which may have been due to process improvement.

The third overtone (210 MHz) parameters of these latter units were also measured, Table 3. Q's were excellent. Unwanted mode plots were not made, but mode scans were visually examined. Unwanted mode performance, while not excellent, is entirely adequate for oscillator applications.

The polishing process used for these 70 MHz resonators resulted in a significant number of scratches which were deepened by the etching process. The polishing step also hid many other forms of surface damage, which were not revealed until the blanks were etched. While the incidence of scratches and other defects was variable from batch to batch, the occurrence was sufficiently frequent to seriously limit device yield. Accordingly, it was decided to attempt to utilize unpolished wafers and, at the same time, to evaluate an alternative polishing process.

2.3.2 100 MHz Fundamental Resonators

To test the feasibility of using unpolished wafers, a group of natural quartz blanks was pinlapped to 29 MHz using 1 micron abrasive, etched to 100 MHz in ammonium bifluoride, plated with 20 mil aluminum electrodes, and sealed in dry nitrogen. The etched surfaces resembled those reported by Vig and coworkers [10] and were essentially scratch-free. Scanning electron micrographs of these surfaces are given in Section 2.5. The mode scan of figure 9 indicates acceptable unwanted mode response typical of the group. Third

TABLE 3

Third Overtone Parameters

70 MHz Fundamental Mode Units

Unit No.	Fs (kHz)	R1 (Ohms)	_	L1/C1 (mH/fF)	`C0/r' (pF/=)	I:Qz m
72	213647.307	138.39	0.239	3.72 0.1490		36 5
73	213501.676	86.03	0.331	2.67 0.2083		42 8
<i>7</i> 5	203129.180	181.35	0.195	3.65 0.1681		26 4
7.7	213409.125	190.93	0.187	3.92 0.1417		28 4
70	213173.800	171.36	0.203	3.09 0.1807		24 4
79	218205.428	206.21	0.176	5.11 0.1040		34 3

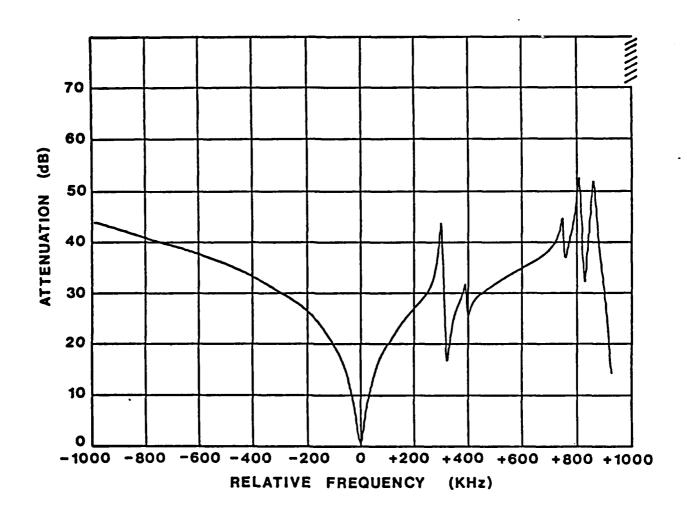


FIGURE 9. TYPICAL UNWANTED MODE RESPONSE, 100 MHz FUNDAMENTAL

overtone mode measurements, however, show multi-moded responses, indicating lack of adequate parallelism. Fundamental mode parameter measurements, Table 4, show 0's below 10,000, considerably below values obtained using polished blanks. The low Q was attributed to the surface finish, although non-parallelism may also have been a factor.

It was felt that while the results obtained using unpolished wafers might be improved somewhat with further effort, their use imposes serious limits on the Q-f product obtainable. Therefore, an alternative polishing process was evaluated. Wafers polished using this process remained essentially free of scratches after etching. A group of 100 MHz fundamental mode resonators was fabricated by etching natural quartz blanks which had been polished using this improved process. The etched units were plated with 20 mil aluminum electrodes and sealed in dry nitrogen. As shown in Table 5, the parameters of these resonators are fairly uniform, indicating the reproducibility of the Q's are typically 25 thousand, with motional resistances as low as 17 ohms. A typical resonance curve and mode plot are shown in figure 10. The motional resistance of the strongest anharmonic mode is approximately 9 times that of the main mode. The closest significant anharmonic is approximately 600 kHz above the main mode. Mode locations are in good agreement with theory.

TABLE 4
Measured Parameters

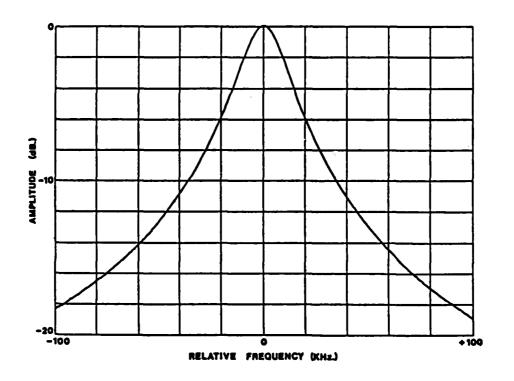
	Fs (kHz)				kū/m
24	100093.148	88.27	0.325	0.91 2.7914	
25	99864.946	71.36	0.371	0.90 2.8081	13 8
26	100195.432	74.95	0.360	0.77 3.2650	17
27	99405.413	55.90	0.425	0.84 3.0447	9 14
28	99512.852	76.06	0.357	0.87 2.9338	7 17
29	99267.189	73 94	0.363	0.91 2.8225	8 21

TABLE 5

Measured Parameters

100 MHz Fundamental Mode Resonators

Unit	Fs	R1	I	L1/C1	CO/r	kQ/m
No.	(kHz)	(Dhms)	(mA.)	(mH/fF)	(pF/-)	
1	100291.684	15.51	0.687	0.66	1.31	27
_		-		3.8414		
4	98898.530	16.60	0.676	0.68	1.29	25
				3.8155	339	75
5 .	97732.634	21.01	0.634	0.73	1.45	21
				3.6549	397	53
6	101283.222	17.52	0.666	0.63	1.46	23
				3.9260	372	61
11	99933.189	14.46	0.698	0.64	1.31	28
				3.9582	331	84
12	102094.946	14.08	0.702	0.63	1.48	29
				3.8455	386	75
13	99537.844	15.80	0.684	0.64	1.29	25
		•		3.9746	326	78
14	101855.475	17.86	0.663	0.64		
				3.7891	392	59
17	98044.975	20.45	0.639	0.70	1.30	21
				3.7455	347	61



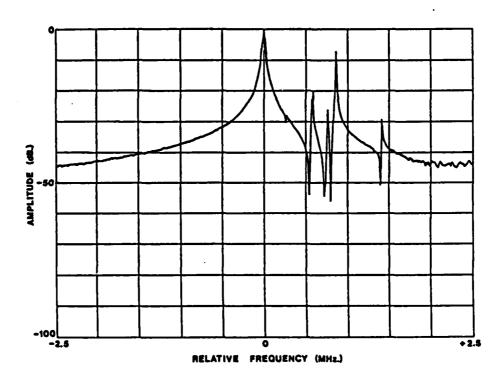


FIGURE 10. FUNDAMENTAL MODE RESPONSE, UNIT NO. 11:

a) RESONANCE CURVE: b) MODE PLOT. RESONANCE
FREQUENCY IS APPROX 99.9 MHz; RES., 14.5 OHMS: Q. 28K
MOTIONAL RESISTANCE OF STRONGEST SPUR IS 130 OHMS

An additional group of 100 MHz fundamental mode resonators was processed using polished natural quartz blanks. These were then plated with 10 mil aluminum electrodes and sealed in vacuum. As seen in Table 6 the 0's of these units ranged up to nearly three times those of the earlier units. The highest Q being 73 thousand, with a motional resistance of 18 ohms. A typical mode plot is shown in figure 11.

The higher Q of these units can be attributed in part to the absence of atmospheric loading. The dissipation factor of a resonator is the sum of a number of terms

$$d = d_{matl} + d_{mech} + d_{atm}$$
 (1)

where d = 1/Q

In Eq. (1) d_{matl} is the intrinsic material loss, estimated for AT-cut quartz by [12]

$$Q_{\text{mat1}} = d_{\text{mat1}} = 1.6 \times 10^7 / f(MHz)$$
 (2)

and d_{atm} represents viscous damping due to the atmosphere.

An appropriate value is given by Bennett [13] as

$$Q_{atm} = d_{atm}^{-1} = 8.55 \times 10^{3} \cdot n/f^{1/2} (MHz)$$
 (3)

for nitrogen at standard temperature and pressure.

In Eq. (1), $d_{\mbox{mech}}$ represents the remaining sources of loss, such as mounting loss.

If we call the resonator Q in vacuum $Q_{\mathbf{v}}$, and in nitrogen, $Q_{\mathbf{N}}$, then

$$Q_N^{-1} = O_v^{-1} + Q_{atm}^{-1}$$
 (4)

TABLE 6 MEASURED PARAMETERS NATURAL QUARTZ 100 MHz FUNDAMENTAL RESONATORS 10 MIL ELECTRODES

Unit No.	Fs (kHz)	R1 (Dhms)	I (inA.)	L1/C1 (mH/fF)	CO/r (pF/-)	kQ/m
1	99977.576	28.22	0.575	2.26 1.1202		50 68
2	100147.884	26.93	0.585	2.24 1.1285	0.83 732	52 71
3	100563.414	21.43	0.630	2.22 1.1308	0.82 727	65 90
4	100491.691	38.74	0.507	2.25 1.1159	0.81 723	
5	100419,380	45.56	0.471	2.25 1.1162	0.84 751	31 42
6	100215.222	37.82	0.512	2.25 1.1193	0.82 733	
7	100174.550	22.92	0.617	2.35 1.072 5	0.79 736	68 88
8	100341.657	21.03	0.626	2.10 1.1977	0.84 700	61 87
9	100213.305	25.58	0.595	2.27 1.1108	0.82 735	56 76
10	100127.630	22.51	0.621	2.30 1.0976	0.83 756	44 85
11	100309.440	52.00	0.441	2.11 1.1954	0.83 691	
12	100387.616	18.01	0.662	2.0B 1.2061	0.84 695	73 105
13	100907.590	20.53	0.638	2.24 1.1104	0.83 743	69 93
14	100232.114	41.25	0.493	2.18 1.1578	0.83 713	
12	99214.000	32.24	0.547	2.43 1.0605	0.82 772	47 61
16	101038.870	50.75	0.414	2.30	0.83 772	25 32

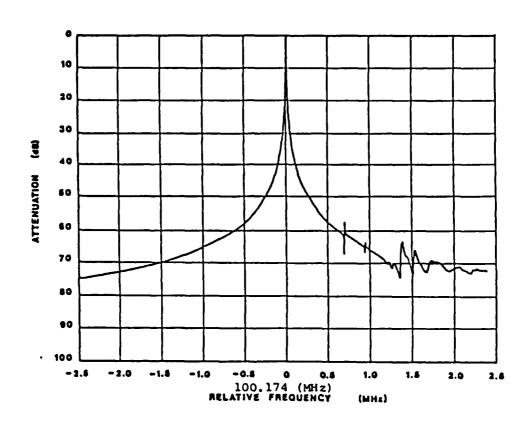


FIG. 11. FUNDAMENTAL MODE RESPONSE
RESONANCE FREQUENCY 100.174 MHz
RESISTANCE 22.9 OHMS; Q, 65K

A few examples show that $Q_{\mathbf{V}}$ can be considerably larger than $Q_{\mathbf{N}}$, especially when d_{mech} is small, so that $Q_{\mathbf{V}}$ approaches Q_{matl} . A "perfect" resonator at 100 MHz would have

$$Q_{v} = Q_{mat1} = 1.6 \times 10^{5}$$

while in dry nitrogen,

 $Q_{atm} = 8.55 \times 10^4$

and

$$Q_N = 5.57 \times 10^4$$

Thus, the Q in vacuum in this ideal case is nearly three times the Q in nitrogen.

To compare the Q's of Table 5 with those of Table 6, the highest Q in Table 5 was 29,000, measured in dry nitrogen. Using Eq. (4), the Q in vacuum is estimated at 44,000, compared with 73,000 for the test resonator in Table 6.

Unless otherwise noted, the data reported in the balance of this report is for resonators sealed in vacuum.

A group of 100 MHz fundamental crystal resonators was fabricated using swept quartz produced at PTI by the electrodiffusion process described in Section 2.4. The results were very encouraging, with the swept cultured quartz appearing nearly as good as the natural quartz in performance.

Table 7 shows the measured parameters for a group of 100 MHz fundamental resonators fabricated out of swept cultured quartz, and figure 12 shows a typical mode response of these units.

TABLE 7

MEASURED PARAMETERS
SWEPT CULTURED QUARTZ

100 MHz FUNDAMENTAL RESONATORS
10 MIL ELECTRODES

Unit No.	Fs (kHz)		_	L1/C1 (mH/fF)		kQ/m
1	100492.431	64.08	0.394	2.21 1.1347	0.84 75á	22 29
2	100485.577	37.77	0.513	2.31 1.0880	0.85 779	39 49
3	100580.415	218.09	0.148	2.28 1.0973		7 9
4	100668.348	26.47	0.588	2.37 1.0552	0.83 784	57 72
5	100454.328	56.09	0.424	2.22 1.1315	0.83 737	25 34
6	100364.451	47.11	0.463	2.24 J.1202	0.86 769	30 39
7	100866.666	67.53	0.383	3.70 0.6723		35 30
8	100314.139	22.95	0.617	2.19 1.1498	0.85 736	60 82
10	100046.687	22.35	0.632	2.14 1.1731	0.87 743	80 81
11	100616.466	21.56	0.829		0.84 764	67 88
12	100456.454	35.72	0.525	2.23 1.1265	0.85 758	37 52

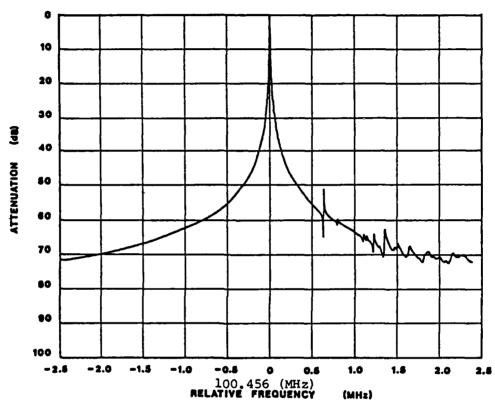


FIGURE 12. FUNDAMENTAL MODE RESPONSE, UNIT NO. 5685-12
SWEPT CULTURED QUARTZ
10 MIL ELECTRODES
RESONANCE FREQUENCY 100.456 MHz;
RESISTANCE 35.7 OHMS; Q, 39K

2.3.3 150 MHz Fundamental Resonators

150 MHz fundamental mode resonators were fabricated using natural quartz blanks polished with the improved polishing technique. Two groups of units were made, one with 5 mil diameter electrodes and a second with 10 mil electrodes, both sealed in vacuum. Table 8 lists the characteristics of the 5 mil group with Q's as high as 49 thousand and motional resistances as low as 34 ohms. Figure 13 shows a typical mode response for one of these units. Table 9 lists the characteristics of the 10 mil group with Q's as high as 38 thousand and motional resistances as low as 17 ohms. Figure 14 shows the mode response for a typical unit with 10 mil electrodes.

2.3.4 250 MHz Third Overtone Resonators

While improvements were being made in the crystal polishing process a group of 250 MHz third overtone resonators were fabricated from natural quartz blanks. These units were plated with 10 mil diameter electrodes, and sealed in vacuum. Table 10 presents measured parameter data, while figure 15 shows a typical resonance curve. The Q's ranged to 47 thousand, with the motional resistance as low as 133 ohms.

TABLE 8

MEASURED PARAMETERS
150 MHz FUNDAMENTAL RESONATORS
5 MIL ELECTRODES

Unit No.	Fs (kHz)	R1 (Ohms)		L1/C1 (mH/fF)		kQ/m
67	150564.051	36.75	0.259	1.74 0.6423		45 39
68	150904.828	74.07	0.181	1.95 0.5694	0.71 1249	25 20
70	151246.858	69.19	0.188	1.93 0.5731	0.63 1101	27 24
71	150586.437	45.41	0.235	1.74 0.6420	0.74 1153	36 31
72	150999.614	39.11	0.252	1.64 0.6764	0.75 1113	40 36
73	150846.994	34.65	0.265	1.80 0.6188		49 42
74	151317.706	33.50	0.269	1.64 0.6728		47 41
75	150927.070	37.25	0.257	1.88 0.5909		48 37
76	150902.120	40.91	0.247	1.73 0.6429	0.74 1147	40 35
78	150601.227	43.34	0.241	1.90 0.5874	0.72	42 34
79	150873.031	37.85	0.256	1.77 0.6302	0.72 1147	44 39

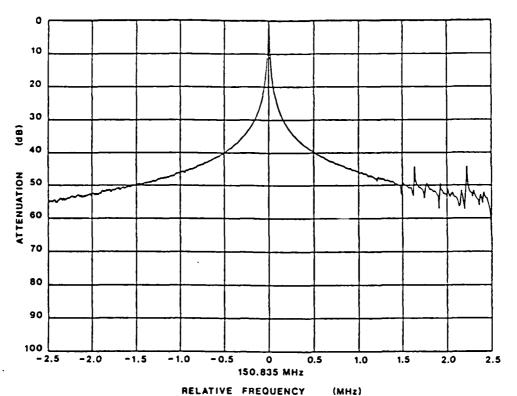


FIGURE 13. FUNDAMENTAL MODE RESPONSE, UNIT NO. 106, 5 MIL ELECTRODES.

RESONANCE FREQUENCY 150.834 MHz;

RESISTANCE 40.3 OHMS; Q, 40K.

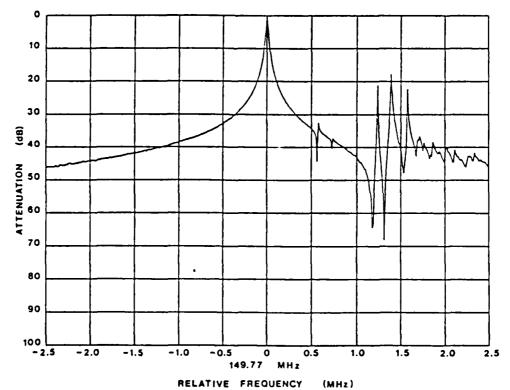


FIGURE 14. FUNDAMENTAL MODE RESPONSE, UNIT NO. 21, 10 MIL ELECTRODES.

RESONANCE FREQUENCY 149.773 MHz;

RESISTANCE 17.4 OHMS; Q, 35K

- 34 -

TABLE 9
MEASURED PARAMETERS
150 MHz FUNDAMENTAL RESONATORS
10 MIL ELECTRODES

Unit No.	Fs (kHz)	R1 (Ohms)	I (mA.)	L1/C1 (mH/fF)	CO/r (pF/-)	kQ/m
1	149925.483	17.31	0.666	0.65 1.7265		
2	150227.586	17.51	0.664		0.98 556	
3	149889.902	21.53	0.626		0.96 540	
4	150217.290	20.11	0.639		0.97 703.	
6	149931.456	20.69	0.634		0.98 574	
7	150048.138	19.60	Ů.644		0.98 564	
9	149921.054	20.93	0.632		0.98 561	
10	149956.026	18.59	Ů.453	0.65 1.7426		
12	150331.898	18.02	0.659	0.64 1.7536		
13	150048.584	22.94	0.614	0.63 1.7832		
14	150588.702	19.03	0.649	0.66 1.6953		33 56

TABLE 10

Measured Parameters

250 MHz Third Overtone Resonators

	Fs (kHz)			L1/C1 (mH/fF)		kQ∕m
2	250603.361	169.37	0.205	4.01 0.1006		37 5
3	250398.829	165.64	0.209	3.89 0.1038	0.88 8526	37 4
4	249990.122	210.96	0.172	3.95 0.1026	0.87 8491	29 3
5	250009.469	247.0B	0.151	4.88 0.0830	ŭ.82 9934	31 3
6	249844.319	140.21	0.237	3.90 0.1039	0.78 7535	44
7	250575.752	200.79	0.179	4.0B 0.0990	ŭ.76 7649	32 4
8	249732.026	193.13	0.185	4.09 0.0993	0.76 7643	33 4
9	250321.100	162.09	0.212	4.04 0.1000		39 5
10	250161.913	150.27	0.225	3.88 0.1043	0.78 7460	41 5
1 1	249890.087	211.14	0.172	4.02 0.1009		
12	249749.840	182.03	0.194	3.99 0.1018		34 4
15	249872.629	141.18	0.235	3.91 0.1038		
16	250889.261	219.07	0.167	4.95 0.0813	0.83 10179	
18	249663.900	133.31	0.245	4.02 0.1011	0.77 7656	47 6

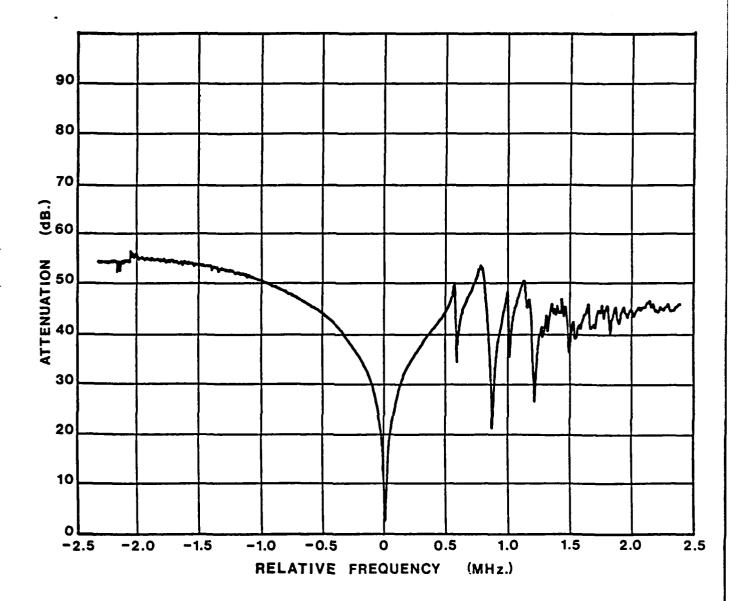


FIGURE 15. THIRD OVERTONE PLOT, UNIT NO. 5
RESONANCE FREQUENCY 250.01 MHz;
RESISTANCE 247 OHMS; Q, 31K

2.3.5 450 MHz Third Overtone Resonators

Using the improved polishing process, two groups of 450 MHz third overtone resonators were fabricated from natural quartz blanks. One group was plated with 5 mil diameter electrodes and a second group was plated with 10 mil diameter electrodes, both groups were sealed in vacuum. Characteristics of representive units are shown in Tables 11 and 12. For the 5 mil electrodes the Q's ranged as high as 27 thousand with the motional resistances as low as 288 ohms. The Q's for the 10 mil group also ranged as high as 27 thousand with motional resistances as low as 100 ohms. Typical mode plots for these two groups are shown in figures 16 and 17.

2.3.6 UHF Fundamental Resonators

In a brief experiment a small group of surplus blanks from another project were etched to a fundamental frequency of approximately 270 MHz, plated with 10 mil aluminum electrodes, and sealed in vacuum. No attempt was made to achieve a uniform frequency. As indicated in Table 13, Q's ranged from 9 thousand to 30 thousand, while mode resistances were between 13 and 30 ohms. Figure 18 is a mode plot for one of the fundamental mode units acceptable unwanted mode levels.

A second group of natural blanks was etched to fundamental frequencies in the range of 250 MHz. These crystals were fabricated into resonators using 5 mil electrodes instead of 10 mil, and were also sealed in vacuum. The results are shown in Table 14 and a mode plot is given in figure 19. Q's ranged from

TABLE 11

MEASURED PARAMETERS
450 MHz THIRD OVERTONE RESONATORS
5 MIL ELECTRODES

Unit No.	Fs (kHz)	R1 (Ohms)	I (mA.)	L1/C1 (mH/fF)	CO/r (pF/-)	kQ/m
67	450258.796	288.42	0.133		0.72 15618	
68	451108.993	356.51	0.111	2.98 0.0418		
70	451814.253	391.02	0.102	3.45 0.0359	0.59 16516	25 2
71	452783.468	964.62	0.044	5.73 0.0216		
72	451543.097	311.69	0.124	2.80 0.0443	0.72 16249	26 2
73	451085.895	308.01	0.126		0.70 15307	
74	452556.329	305.69	0.127		0.73 15304	
75	451149.348	318.04	0.122	3.00 0.0415	0.73 17647	27 2
76	451143.983	332.01	0.118		0.70 17249	
78	450156.550	486.02	0.084		0.69 16055	
79	451152.917	299.34	0.129		0.68 15245	26 2
80	451698.208	326.92	0.119	2.73 0.0455		
84	452765.978	310:05	0.125	2.85 0.0433	0.70 16194	26 2

TABLE 12

MEASURED PARAMETERS
450 MHz THIRD OVERTONE RESONATORS
10 MIL ELECTRODES

Unit No.				L1/C1 (mH/fF)		kQ/m
1	449780.808	99.55	0.301	0.92 0.1357		26 4
, 2	450687.818	110.89	0.280	1.01 0.1229		26 3
3	449661.861	109.81	0.282	0.87 0.1437	0.97 6741	3 22
4	449942.578	573.99	0.072	1.42 0.0882		7 1
5	456263.222	149.45	0.226	1.40 0.0870		
6	449782.015	115.18	0.272	1.04 0.1205		
7	450147.997	113.40	0.275	1.03		
9	449792.793	101.84	0.296	0.96 0.1299	0.98 7580	27 4
10	449895.557	102.61	0.295	0.96 0.1299		
11	449659.175	104.76	0.291	0.93 0.1350	0.98 7265	25 3
12	450963.726	124.03	0.259	1.05 0.1186		
13	450153.754	125.84	0.256	1.03 0.1216	0.99 8178	23 3
14	451662.245	122.43	0.261	1.14 0.1089		26 3

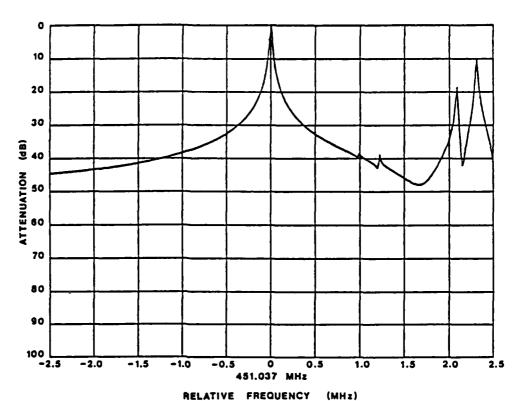


FIGURE 16. THIRD OVERTONE RESPONSE, UNIT NO. 106, 5 MIL ELECTRODES.

RESONANCE FREQUENCY 451.04 MHz;
RESISTANCE 323.0 OHMS; Q, 25K

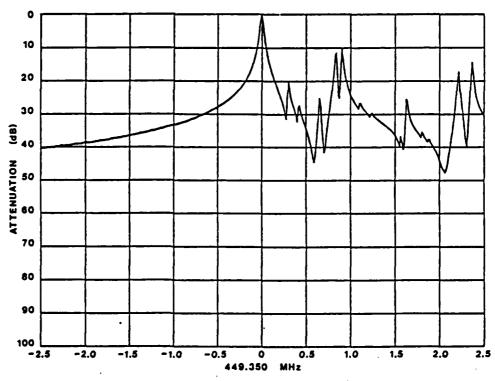


FIGURE 17. THIRD OVERTONE RESPONSE, UNIT NO. 21, 10 MIL ELECTRODES.
RESONANCE FREQUENCY 449.35 MHz;
RESISTANCE 120 OHMS; Q, 23K.

TABLE 13
Measured Parameters

250 MHz Fundamental Mode Resonators

Unit No.	Fs (kHz)	R1 (Ohms)	-	LI/CI (mH/fF)		kQ/m
1	261928.600	29.65	0.565	0.16 2.2456	1.12 500	9 18
2	274645.244	13.11	0.713	•	1.33 581	19 33
3	263392.211	24.86	0.601	0.17 2.2110	1.28 578	11 19
4	252341.306	15.03	0.692	0.23 1.6984	1.40 825	25 30
6	269220.440	13.16	0.712	0.14 2.4122	1.29 537	19 35

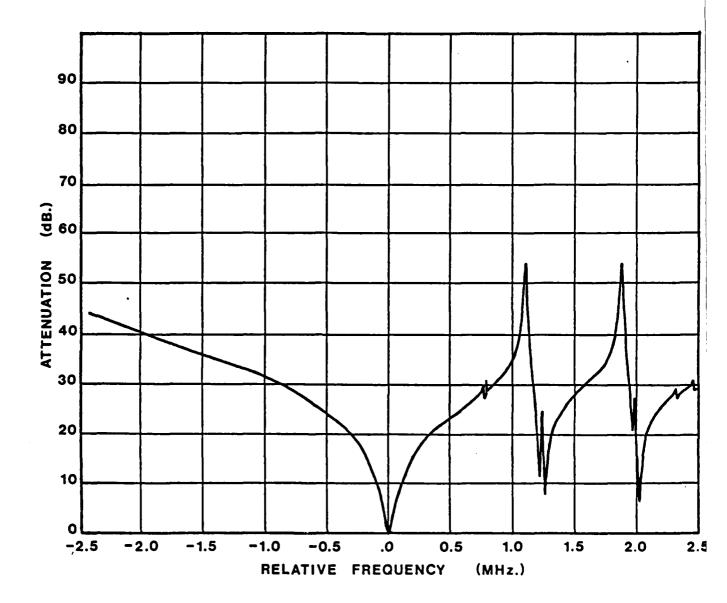


FIGURE 18. FUNDAMENTAL MODE PLOT, UNIT NO. 6
RESONANCE FREQUENCY 269.22 MHz;
RESISTANCE 13.2 OHMS; Q, 19K

TABLE 14

MEASURED PARAMETERS
250 MHz FUNDAMENTAL RESONATORS
5 MIL ELECTRODES

Unit No.	Fs (kHz)			L1/C1 (mH/fF)		kQ/m
9	256409.894	43.38	0.482	0.72 0.5344		27 15
10	245383.418	68.10	0.381	0.81 0.5197		
12	244842.908	50.31	0.449	0.66 0.6400		
13	246772.969	37.72	0.513	0.68 0.6131	0.75 1226	
15	244889.166	37.19	0.516	0.73 0.5792	0.73 1266	
16	246710.000	94.75	0.311	0.86 0.4825		14
17	245651.796	60 . 99	0.405	0.82 0.5093		
18	246728.707	61-67	0.403	0.82 0.5087		
67	234241.803	83.03	0.338	0.72 0.6432		
68	250227.323	42.31	0.487	0.73 0.5573		
78	256307.014	59.60	0.411	0.54 0.7181	0.79 1094	15 13

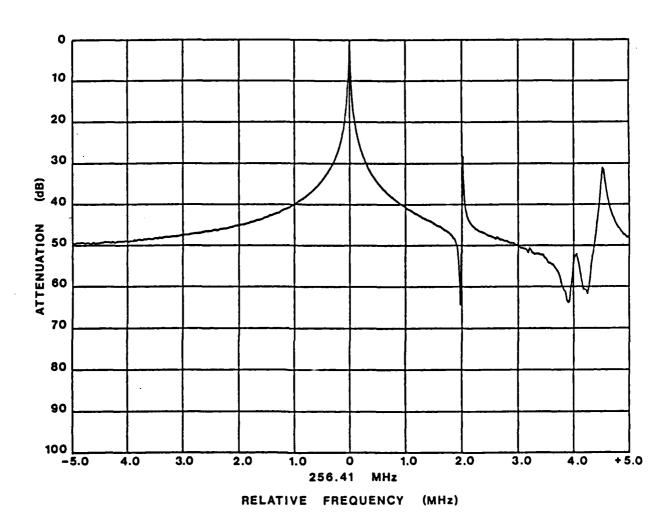


FIGURE 19. FUNDAMENTAL MODE RESPONSE, UNIT NO. 9
RESONANCE FREQUENCY 256.41 MHz;
RESISTANCE 43.4 OHMS; Q, 27K.

14 thousand to 30 thousand, slightly higher than the units made with 10 mil electrodes. Comparing these units with the third overtone units shown earlier in Table 10, it is seen that, as would be expected, the resistance and 0 of the fundamental mode devices are both lower than for the third overtone units.

The 250 MHz third overtone and 250 MHz fundamental units illustrate a fundamental design trade-off: for a given frequency and electrode configuration, the motional impedance level is proportional to the cube of the overtone. Thus the motional inductance of the third overtone 250 MHz units is approximately 27 times that of the 250 MHz fundamental ones. However, and typically, the Q of the fundamental units is less than that of the overtone units so that motional resistances are in a ratio of very roughly 1 to 10, and of course the capacitance ratio of the fundamental is much lower than for the third overtone — in this instance by a factor of 12 or 15.

In general, for oscillator applications, fundamental mode resonators are favored where ease of frequency "pulling" is important; e.g., temperature-compensated and voltage-controlled oscillators. In some precision oscillator applications, however, just the opposite is wanted in order to minimize the effects of oscillator circuitry on frequency, and hence on aging. The use of overtones also favors good resonator aging. Moreover, for low phase noise and good short-term stability it is desirable to maximize Q -- specifically, the loaded O of the resonator in the oscillator circuit. The use of very high overtones, even in precision oscillators, is, however, limited by practical impedance level consideration and the need for enough pullability to correct for inevitable manufacturing tolerances in resonator frequency.

To further explore the possibility of producing UHF range fundamental resonators, two groups of blanks were etched to approximately 350-525 MHz on the fundamental. These units were plated with 5 mil electrodes and sealed in vacuum. Their characteristics are shown in Tables 15 and 16. Figure 20 shows a typical fundamental mode response for one of these units. Q's for resonators around 500 MHz ranged up to 13 thousand and mode resistances were as low as 48 ohms. It should be noted that no attempt was made to trim all the blanks to any particular frequency. A thickness difference between blanks of 1 micron at 30 MHz represents a frequency difference of 550 kHz, while the same 1 micron difference at 500 MHz represents a frequency difference of 215 MHz. Thus a large variation in frequency between individual units within each UHF group is not surprising.

Encouraged by these preliminary results, two more groups of natural quartz blanks were etched to UHF fundamental frequencies.

The first of these two groups was plated with 5 mil electrodes and sealed in vacuum. The resulting resonators ranged in frequency from approximately 440 MHz to 674 MHz, as seen in Table 17. A mode plot of one of the higher units is shown in figure 21.

The second group of blanks was etched, plated with 2.5 mil electrodes, and sealed in vacuum. The fundamental frequencies of this group ranged from approximately 700 MHz to over 1.6 GHz. Table 18 shows a portion of these resonators, with the highest being 954 MHz. The automatic test equipment used for these measurements has an upper frequency limit of 1000 MHz. Unit #5 was measured on a manual system, and showed a frequency of 1,655 MHz. Figure 22 shows a mode plot of this unit, while figures 23 and 24 show mode plots of an 823 MHz and an 954 MHz resonator respectively.

TABLE 15

Measured Parameters

UHF Fundamental Mode Resonators

Unit No.	Fs (kHz)		_	L1/C1 (mH/fF)	kQ/m
1	434941.835	45.40	0.467	0.25 0.5334	 15 5
. 18	503400.551	48.47	0.457	0.20 0.5049	 13 4

TABLE 16

Measured Parameters

UHF Fundamental Mode Resonators

Unit No.	Fs (kHz)	R1 (Ohms)	I (mA.)	L1/C1 (mH/fF)	C0/r (pF/-)	kQzm
2	476210.880	67. 43	0.383	0.20 0.5481	1.71	9
3	361237.326	39.46	0.503	0.27 0.7314	1.57 2146	15
6	465355.502	33.52	0.539	0.13 0.8873	1.74 1963	11
7	401158.368	47.17	0.463	0.22 0.7149	1.62 2268	12
9	437209.312	38.51	0.508	0.17 0.7791	1.66	12
11	527605.848	51.93	0.441	0.12 0.7891	0.90 1139	7 6

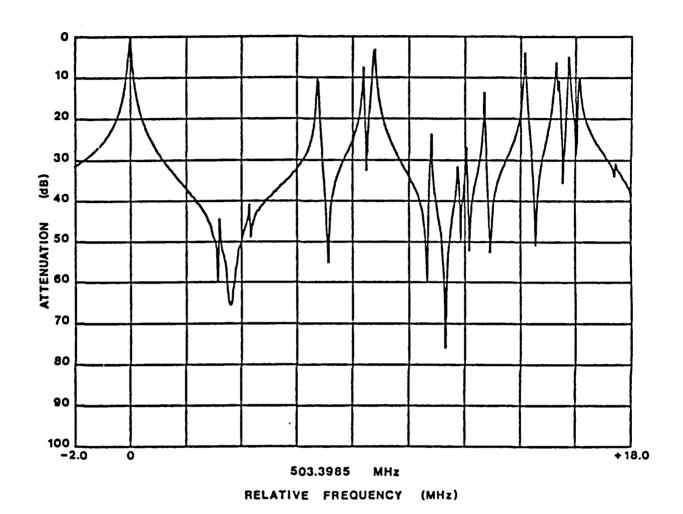


FIGURE 20. FUNDAMENTAL MODE RESPONSE, UNIT NO. 18
RESONANCE FREQUENCY 503.4 MHz;
RESISTANCE 48 OHMS; Q, 13K

TABLE 17

Measured Parameters

UHF Fundamental Mode Resonators

Unit No.	Fs (kHz)			L.17C1 (mHZFF)		1. C! / m
34	610116.828	52.29	0.440	0.09 0.7629		7 3
35	547929.000	32.17	0.548	0.10 0.8786		
36	440305.616	30.65	0.558	0.29 0.4547		26 7
37	518425.784	81.93	0.341	0.13 0.7064		চ ব
38	572207.732	39.26	0.504	0.09 0.8163		9 4
41	674181.290	53.89	0.400	0.05 1.0729	2.02 1887	4 2
42	665416.322	1292.17	0.034	2.24 0.0156		7 O
43	575214.867	73.97	0.043	0.10 0.7431		5 2
44	578758.996	125.27	0.257	0.15 0.5030		4 1
45	602772.230	61.37	0.404	0.09 0.7948	1.87 2348	5
46	620121.079	00.22	0.541	0.08 0.8759	1.91 2185	<i>फ़</i> च
47	513029.516	43.39	0.482	0.10 0.9214		6

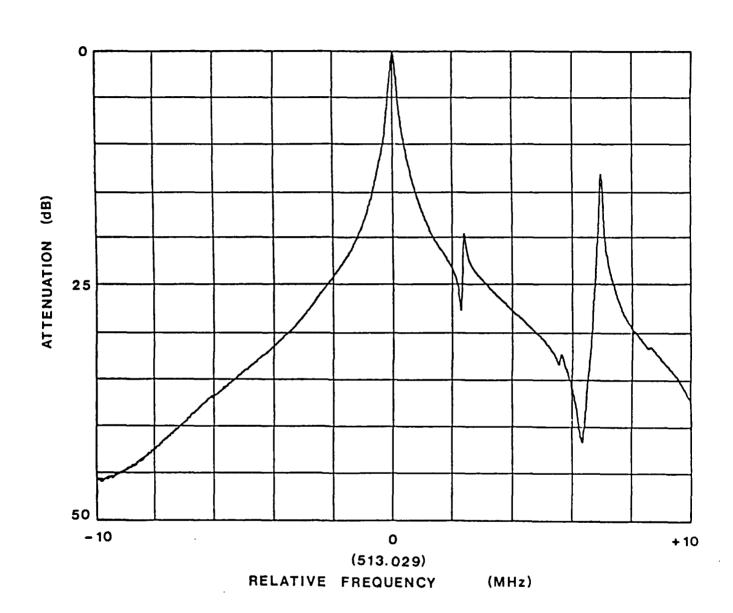


FIGURE 21. FUNDAMENTAL MODE PLOT, UNIT NO. 47
RESONANCE FREQUENCY 513.029 MHz;
RESISTANCE 43.4 OHMS; Q. 8 K

TABLE 18

Measured Parameters

UHF Fundamental Mode Resonators

Unit No.	Fs (kHz)	R1 (Ohms)	I (mA.)	L1/C1 (mH/fF)	CO/r (pF/-)	kQ/m
1	843145.260	27.65	0.580	0.03 1.1814	1.17 992	င်
2	735172.944	58. 30	0.415	0.08 0.5904	1.11 1872	6 3
4	823007.394	29.76	0.564	0.04 1.0149	1.07 1054	6 6
7	717249.937	75.75	0.358	0.07 0.7372	0.91 1236	4 3
7	954273.825	29.85	0.564	0.02 1.1461	1.02 892	5 5

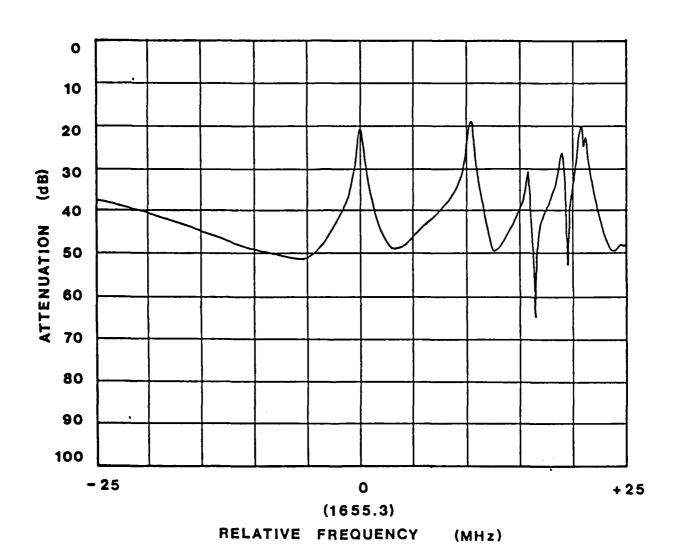


FIGURE 22. FUNDAMENTAL MODE PLOT, UNIT NO. 5 RESONANCE FREQUENCY 1655.3 MHz.

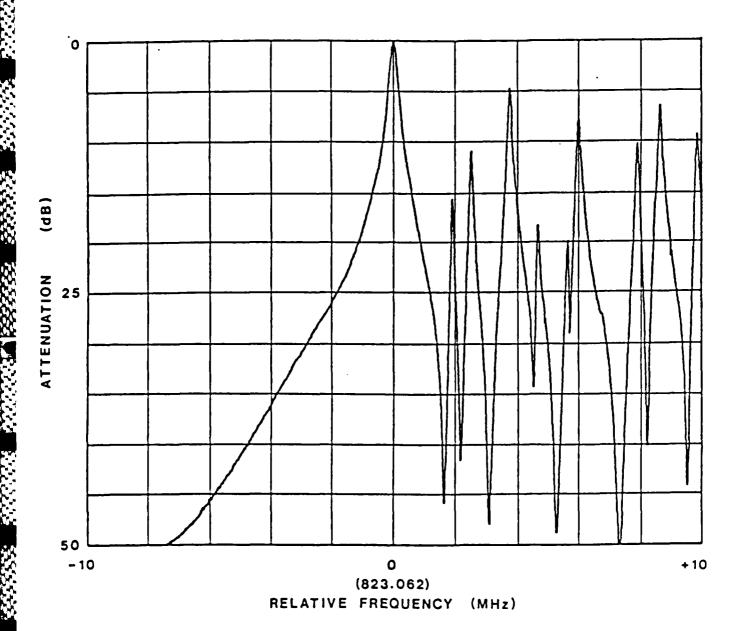


FIGURE 23. FUNDAMENTAL MODE PLOT, UNIT NO. 4
RESONANCE FREQUENCY 823.062 MHz;
RESISTANCE 29.8 OHMS; Ω , 6K

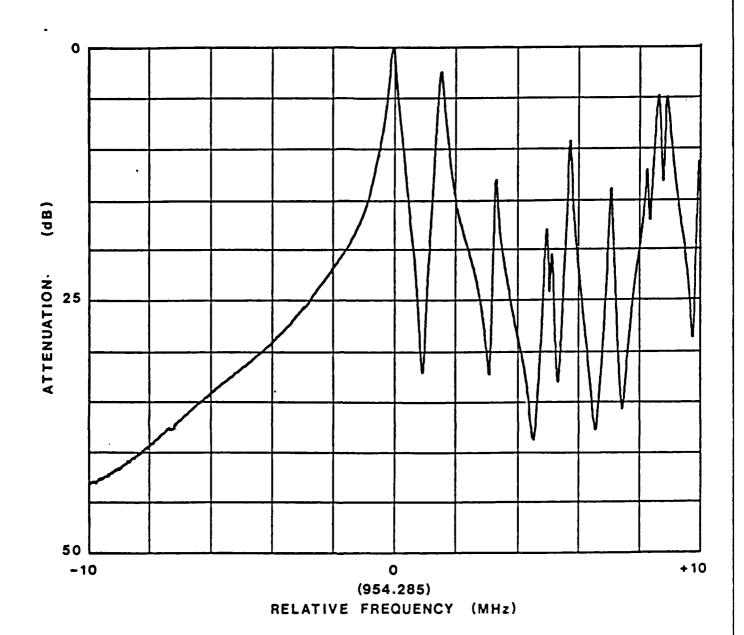


FIGURE 24. FUNDAMENTAL MODE PLOT, UNIT NO. 9
RESONANCE FREQUENCY 954.285 MHz
RESISTANCE 29.8 OHMS; Q, 5K

These higher frequency resonators exhibit larger than normal spurious mode responses. The electrode dimensions and mass loading used for these crystals were not optimum for the frequency. Different techniques from those currently in use would have to be developed to achieve optimum electrode configurations at frequencies in the gigahertz range. The highest unelectroded blank frequency measured was over 1.7 GHz. The membrane thickness required to achieve this frequency is just under 1.0 microns. The fact that these blanks survived all the processing steps necessary to produce finished units testifies to the inherent strength of the quartz membrane.

2.3.7 Oscillator Circuit

To demonstrate the feasibility of using the VHF units fabricated under this program, a simple bread-board 100 MHz crystal oscillator was constructed, figure 25. The circuit was then built in prototype form using standard printed-circuit construction. It consists of a single-transistor oscillator stage followed by an output buffer, and will function to 150 MHz.

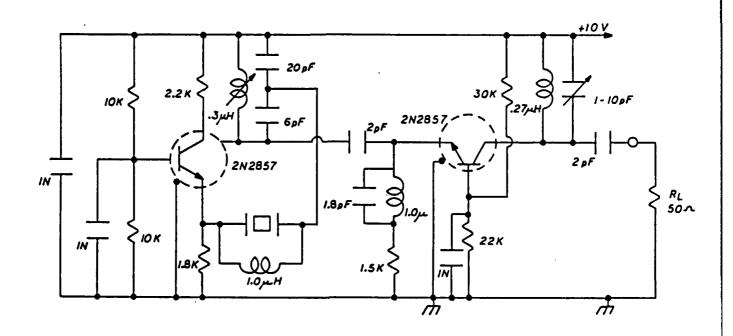


FIGURE 25. CIRCUIT DIAGRAM, PROTOTYPE 100-150 MHz OSCILLATOR

2.4 Electrodiffusion of Quartz

It had previously been found that all attempts made at producing VHF resonators by wet chemical etching of cultured quartz resulted in severely degraded devices which were unusable. Two phenomena are responsible for this degradation. One is an elongated "etch pit" which appears on the surface of the blank. Figure 26 is a scanning electron microscope photograph (150x) of the surface of a cultured quartz blank after etching to approximately 70 MHz. Typical dimensions for an etch pit after etching away 30 microns of material are 65 microns long, 22 microns wide, and 4 microns deep.

The second phenomenon is a small tubular channel etched completely through the thickness of the crystal blank and connecting an etch pit on one surface of the blank to a corresponding etch pit on the opposing surface. These have been described in the literature as "etch channels" or "etch tunnels", and are typically 1.25 microns in diameter after etching 30 microns. Figure 27 is an optical microscope photograph (105x) of a deeply etched cultured quartz blank with numerous pairs of elongated etch pits visible. Each pair is made up of an etch pit on the top surface, connected to a corresponding etch pit visible on the bottom surface by an etch channel, visible as a dark line in the photograph. Figure 28 is another optical microscope photograph (262.5x) showing the same phenomenon at higher magnification. Again the photograph shows pairs of etch pits, one each on both the top and bottom surface of the quartz membrane, each pair connected by an etch channel which penetrates completely through the blank.

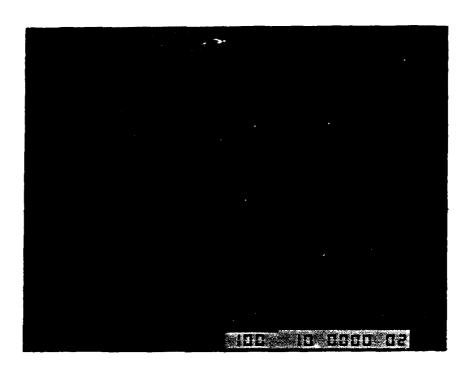


FIGURE 26. CULTURED QUARTZ, ETCHED 30 MICROMETERS SEM (150x)



FIGURE 27. CULTURED QUARTZ, ETCHED TO 70 MHz
OPTICAL (105x)



FIGURE 28. CULTURED QUARTZ, ETCHED TO 70 MHz OPTICAL (262.5x)

Both types of defects result in either device failure, as holes are generated in the membrane, or severe degradation of the electrical performance. It has been observed that not all etch pits have etch channels, but that all observed etch channels begin and end at opposing etch pits. Typically there are many more etch pits than etch channels observable after etching.

Natural quartz has been used for the majority of the chemical etching performed under this contract in order to reduce the incidence of etch channels and pits. Cultured quartz can be post-processed by electrodiffusion ("sweeping".) Using suitably swept cultured quartz, etch channels can be almost entirely eliminated [14]. With this technique, selected ions are made to migrate under the influence of high temperature and electric fields toward one face of a bar of quartz. This highly contaminated surface is then removed, leaving a quartz bar relatively free of impurities.

Because of the high cost and limited availability of commercially swept quartz, it was decided to construct a small sweeping facility. During the term of this contract three groups of cultured quartz bars were swept in an electrodiffusion process. Only the first lot was processed into crystal wafers and fabricated into final devices. During sweeping, the quartz was brought from room temperature to 525°C over a twenty-four hour period. It was maintained at 525°C with an electrical field of 1500 volts per centimeter of thickness for a period of seven days. The temperature was then slowly lowered to room temperature with the field still applied over a second twenty-four hour period.

Both swept and unswept material from the same lot was fabricated into polished blanks approximately 55 microns thick. The blanks were then etched 32 microns under the same conditions and inspected visually for etch channel density. The unswept blanks exhibited a mean etch channel density of 481 channels per square centimeter with a sigma value of 298 channels. The mean etch channel density for the swept blanks was 2.1 with a sigma value of 2.5. While all the unswept blanks had etch channels, half of the swept blanks had no etch channels at all.

The work performed to date with swept cultured quartz indicates that it may no longer be necessary to use natural quartz in the production of VHF and UHF fundamental crystal resonators. This would allow better control over the quality of the starting material and favorably improve the consistency and repeatability of future VHF crystal production.

2.5 Surface Finish Effects

Lapping crystals with an inert alumina lapping abrasive is a purely mechanical process, but the polishing of quartz crystals is usually done with media containing a chemically reactive rare earth oxide such as cerium dioxide. This technique combines mechanical abrasion with chemical dissolution and the resulting finish is often subject to hidden surface damage. An amorphous silica layer often covers scratches which only become visible after chemical etching.

An attempt was made to fabricate VHF resonators out of natural quartz blanks which had been mechanically lapped with a 1 micron aluminum oxide abrasive in lieu of polishing. The blanks were etched to 100 MHz fundamental frequency and made into the resonators described in Section 2.3.2. The etched surfaces are too microscopically rough to allow the observation of Haidinger's fringes under monochromatic light, but are essentially scratch-free. highest Q observable on these units was 9 thousand and the lowest motional resistance was 55.9 ohms. Units which were made from carefully polished blanks had maximum 0's of 73 thousand and motional resistances as low as 14.5 ohms. Figure 29 shows an SEM(500x) photograph of the etched surface finish of a lapped crystal. An undulating surface can be seen, which remains even with further etching. Figure 30 shows an SEM(500x) photograph of the etched surface finish of a polished crystal. It is believed the observable surface differences are the main reason for the Q degradation on lapped blanks fabricated into VHF resonators. Figure 31 compares measured values of Q for VHF and UHF resonators fabricated from polished blanks with the most probable

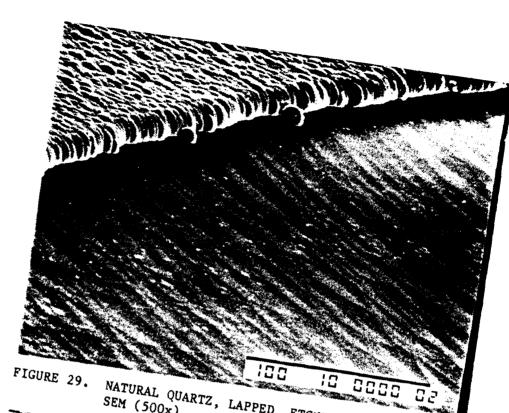


FIGURE 29. NATURAL QUARTZ, LAPPED, ETCHED TO 100 MHz,

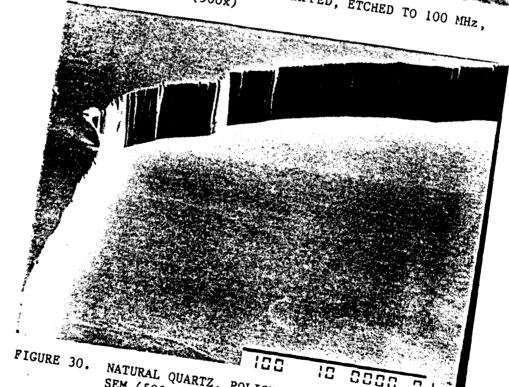


FIGURE 30. NATURAL QUARTZ, POLISHED, ETCHED TO 250 MHz,

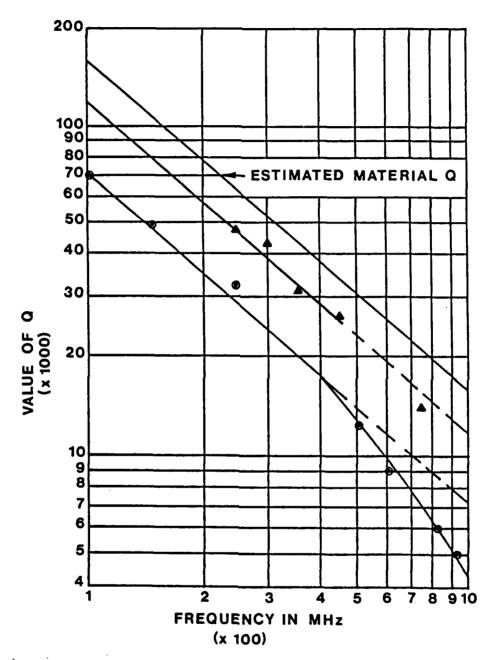


FIGURE 31. MEASURED VALUES OF Q FOR FUNDAMENTAL (⊙) AND THIRD OVERTONE (♠) AT-CUT RESONATORS

material Q [12] over a wide range of frequencies.

Third overtone measurements on the lapped-only blanks show multi-moded responses, indicating lack of adequate parallelism. This is primarily due to the lack of easy methods of determining blank parallelism of lapped surfaces, rendering it impractical to monitor this aspect of the lapping process.

Polished blanks, on the other hand, can easily be checked for parallelism by oberving self-interference fringes under a monochromatic light. Although the lapped blanks are still suitable for many resonator applications, the superior performance of polished blanks led to their being used predominantly for this contract work.

2.6 Summary Of Results

Using wet chemical etching of polished, AT-cut quartz wafers, resonators have been fabricated at fundamental frequencies from 70 MHz to 1.6 GHz. In figure 31 the best Q's achieved experimentally are compared with the estimated material Q. Bearing in mind that electrode designs have not been optimized, especially at the higher frequencies, these results are extremely encouraging.

Sweeping has successfully been used to produce cultured quartz which is nearly free of etch channels. This material has been used to fabricate 100 MHz resonators with good results.

Finally, to demonstrate the feasibility of using chemically milled resonators in oscillators, a simple breadboard circuit, limited to 150 MHz by transistor parameters, was built. Using suitable transistors and UHF construction practice, the frequency range can be extended, but such activity was beyond the scope of this program.

3 Conclusion

High fundamental frequency AT-cut resonators are potentially useful for VHF, UHF, and microwave frequency generation as well as for VHF and UHF crystal filters. The work performed to date has demonstrated the feasibility of using wet chemical etching techniques to fabricate resonators at fundamental frequencies into the gigahertz range. The processes used lend themselves to practical manufacturing use. The work performed to date with swept cultured quartz indicates that it can be used for the production of VHF and UHF fundamental crystal resonators by etching. This will allow better control over the quality of the starting material and improve the consistency and repeatability of future VHF crystal production.

Although the feasibility has been demonstrated and practical devices have been made, there remain several areas for future work. These include improved methods of final frequency adjustment, aging studies, the use of plasma etching for wafer frequency adjustment, and phase noise and acceleration sensitivity studies.

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